

# Laboratory Procedures

## Experiment 1

### Process Objectives

- To observe proper safety technique with all laboratory equipment.
- To use laboratory apparatus skillfully and efficiently.

### Learning Objectives

- To learn the names and functions of all the apparatus in the laboratory.
- To develop a positive approach toward laboratory safety.

### Introduction

The best way to become familiar with chemical apparatus is to actually handle the pieces yourself in the laboratory. This experiment is divided into several parts in which you will learn how to adjust the gas burner, insert glass tubing into a rubber stopper, use a balance, handle solids, measure liquids, filter a mixture, and measure temperature and heat. Great emphasis is placed on safety precautions that should be observed whenever you perform an experiment and use this apparatus. Several useful manipulative techniques are also illustrated on pages 9 through 11 under the heading Techniques and Safety Sketches. In many of the later experiments, references will be made to these Techniques and Safety Sketches. You will also be referred to many of the safety precautions and procedures explained in all parts of this experiment. It is important that all students develop a positive approach to a safe and healthful environment in the laboratory.

### Safety



Take the necessary safety precautions before beginning each part of this experiment. Wear safety goggles, apron, and gloves. Get into the "good habit" of always putting on this standard safety equipment as soon as you enter the lab. It is important that you and your partner observe all safety precautions while conducting experiments. See pages 3 through 8. Read all safety cautions noted in your procedures and discuss them with your teacher.

### Part 1 The Burner

#### Apparatus

heat-resistant mat  
forceps  
sparker

burner and tubing  
evaporating dish

#### Materials

copper wire, 18 gauge

#### Recording Your Observations

Record your observations in the data tables that you make in your notebook. Also, answer all questions in your notebook.

#### Procedures

1. The Bunsen or Tirrell burner is commonly used as a source of heat in the laboratory. Although the details of construction vary among burners, each has a gas inlet located in the base, a vertical tube or barrel in which the gas is mixed with air, and adjustable openings or ports in the base of the barrel. These ports admit air to the gas stream. The burner may have an adjustable needle valve to regulate the flow of gas. In some models the gas flow is regulated simply by adjusting the gas valve on the supply line. The burner is always turned off at the gas valve, never at the needle valve. Look at Figure 1-1 as you examine your Bunsen burner and locate these parts.

**CAUTION** Before you light the burner, check to see that you and your partner have taken the following safety precautions against fires: Wear safety goggles, aprons, and gloves. Confine long hair and loose clothing: Tie long hair at the back of the head and away from the front of the face, roll up long sleeves on shirts, blouses, and sweaters away from the wrists. You should also know the locations of fire extinguishers, fire blankets, safety showers, and sand buckets and how to use them in case of a fire.

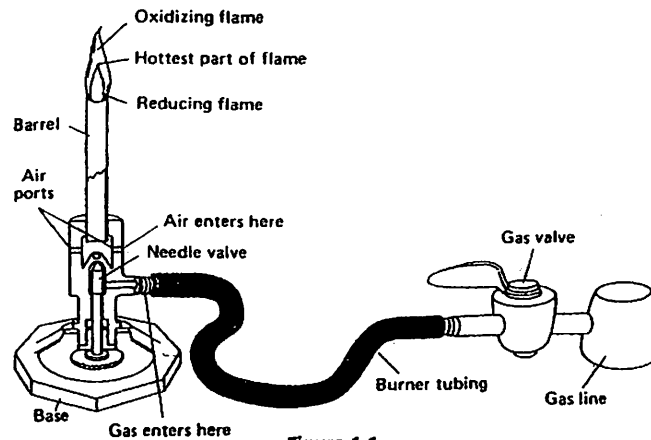


Figure 1-1

2. In lighting the burner, partially close the ports at the base of the barrel, turn the gas full on, hold the sparker about 5 cm above the top of the burner, and proceed to light. The gas flow may then be regulated by adjusting the gas valve until the flame has the desired height. If a very low flame is needed, remember that the ports should be partially closed when the gas pressure is reduced. Otherwise the flame may burn inside the base of the barrel. When improperly burning in this way, the barrel will get very hot, and the flame will produce a poisonous gas, carbon monoxide.

**CAUTION** Carbon monoxide is a poisonous gas. If the flame is burning inside the base of the barrel, immediately turn off the gas at the gas valve. Do not touch the barrel, for it is extremely hot! Allow the barrel of the burner to cool off and then proceed as follows:



Begin again, but first decrease the amount of air admitted to the burner by partly closing the ports. Turn the gas full on and then relight the burner. Control the height of the flame by adjusting the gas valve. By taking these steps, you should acquire a flame that is burning safely and is easily regulated throughout the experiment.

3. Once you have a flame that is burning safely and steadily, you can experiment by completely closing the holes (ports) at the base of the burner. What is the result?

Using the forceps, hold an evaporating dish in the tip of the flame for about three minutes. Place the dish on a heat-resistant mat, allow the dish to cool, and then examine its underside. Describe the results and suggest a possible explanation.

Such a flame is seldom used in the laboratory. For laboratory work, you should adjust the burner so that the flame will be free of yellow color, nonluminous, and also free from the "roaring" sound caused by admitting too much air.

4. Regulate the flow of gas to give a flame extending roughly 8 cm above the barrel. Now adjust the supply of air until you have a quiet, steady flame with a sharply defined, light blue inner cone. This adjustment gives the highest temperature possible with your burner. Using the forceps, insert a 10-cm piece of copper wire into the flame just above the barrel. Lift the wire slowly up through the flame. Where is the hottest portion of the flame located? Hold the wire in this part of the flame for a few seconds. What is the result?
5. Shut off the gas burner. Now think about what you have just observed in Procedures 3 and 4. Why is the nonluminous flame preferred over the yellow luminous flame in the laboratory? (Hint: The melting point of copper is 1083°C.)
6. At the end of this part of the experiment, all the equipment you store in the lab locker or drawer should be completely cool, clean, and dry. It should also be arranged in an orderly fashion for the next lab experiment. Check to see that the valve on the gas jet is completely shut off. Remember that hands should be washed thoroughly with soap at the conclusion of each laboratory period.

**CAUTION** In many experiments you will have to dispose of a liquid chemical at the end of a lab. Always ask your teacher for the correct method of disposal. In many instances liquid chemicals can be washed down the sink's drain by diluting them with plenty of tap-water. Very toxic chemicals should be handled only by your teacher. All apparatus should be washed, rinsed, and dried.



7. Remember to wash your hands thoroughly at the end of this part of the experiment.

## Part 3 Handling Solids

### Apparatus

test tube  
glazed paper

porcelain or plastic spoon  
(or spatula)

### Materials

sodium chloride

### Procedures

1. Solids are usually kept in wide-mouthed bottles. A porcelain or plastic spoon (or spatula) should be used to dip out the solid. See Figure 1-3.

**CAUTION** Do not touch chemicals with your hands. Some chemical reagents readily pass through the skin barrier into the bloodstream and can cause serious health problems. Some chemicals are extremely corrosive. Always wear gloves, apron, and safety goggles when handling chemicals. Carefully check the label on the reagent bottle or container before removing any of the contents. Never use more of a chemical than directed. You should also know the locations of the lab shower and eyewash and how to use them in case of an accident.



2. Remove a spoonful of sodium chloride from its reagent bottle. In order to transfer the sodium chloride to a test tube, first place it on a piece of glazed paper about 10 cm square. Roll the paper into a cylinder and slide it into a test tube that is lying flat on the table. When you lift the tube to a vertical position and tap the paper gently, the solid will slide down into the test tube. See Figure 1-4.

**CAUTION** Never try to pour a solid from a bottle into a test tube. As a precaution against contamination, never pour unused chemicals back into their reagent bottles.



3. Throw the solid sodium chloride and glazed paper into the waste jars or containers provided by your teacher.

**CAUTION** Never discard chemicals or broken glassware in the wastepaper basket. This is an important safety precaution against fires, and it prevents personal injuries (such as hand cuts) to anyone who empties the wastepaper basket.



4. Remember to clean up the lab and wash your hands thoroughly at the end of this part of the experiment.

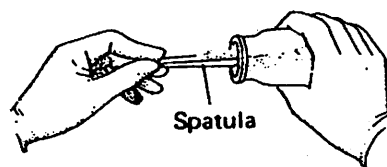


Figure 1-3

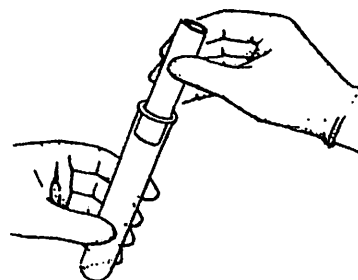


Figure 1-4

## Part 5 Measuring Liquids

### Apparatus

graduated cylinder  
buret clamp  
buret

pipette  
ring stand  
beakers, 50 mL, 250 mL

### Materials

water

### Procedures

1. For approximate measurements of liquids, a graduated cylinder such as the one shown in Figure 1-6 is generally used. These cylinders are usually graduated in milliliters (mL). Such a graduated cylinder may read from 0 to 10 mL, 0 to 25 mL, or more, from bottom to top. It may also have a second row of graduations reading from top to bottom. Examine your cylinder for these markings.

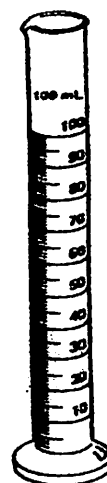


Figure 1-6

## Part 6 Filtration

### Apparatus

ring stand  
stirring rod  
ceramic-centered wire gauze  
evaporating dish  
marker  
wash bottle

filter paper  
iron ring  
burner and tubing  
two beakers, 250 mL  
funnel

### Materials

sodium chloride  
water

fine sand

### Procedures

1. Sometimes liquids contain particles of insoluble solids, present either as impurities or as precipitates formed by the interaction of the chemicals used in the experiment. If the particles are denser than water, they soon sink to the bottom. Most of the clear, supernatant (swimming above) liquid may be poured off without disturbing the precipitate. Such a method of separation is known as decantation. See page 9 for the proper techniques.

2. Fine particles, or particles that settle slowly are often separated from a liquid by filtration. Support a funnel on a small ring on the ring stand as shown in Figure 1-12. Use a beaker to collect the filtrate. Adjust the funnel so that the stem of the funnel just touches the inside wall of the beaker.

3. Fold a circular piece of filter paper along its diameter, and then fold it again to form a quadrant. See Figure 1-13. Separate the folds of the filter, with three thicknesses on one side and one on the other; then place in the funnel. The funnel should be wet before the paper is added. Use your plastic wash bottle. Then wet the filter paper with a little water and press the edges firmly against the sides of the funnel so no air can get between the funnel and the filter paper while the liquid is being filtered. **EXCEPTION:** A filter should not be wet with water when the liquid to be filtered does not mix with water. Why?

4. Dissolve 2 or 3 g of salt in a beaker containing about 50 mL of water, and stir into the solution an equal bulk of fine sand. Then filter out the sand by pouring the mixture into the filter, observing the following suggestions:

a. The filter paper should not extend above the edge of the funnel. It is better to use a filter disc that leaves about 1 cm of the funnel exposed.

b. Do not fill the filter. It must never overflow.

c. Try to establish a water column in the stem of the funnel, thus excluding air bubbles, and then add the liquid just fast enough to keep the level about 1 cm from the top of the filter.

d. When a liquid is poured from a beaker or other container, it may adhere to the glass and run down the outside wall. This may be avoided by holding a stirring rod against the lip of the beaker, as shown in Figure 1-12. The liquid will run down the rod and drop off into the funnel without running down the side of the beaker.

The sand is retained on the filter paper. What property of the sand enables it to be separated from the water by filtration? What does the filtrate contain?

5. The salt can be recovered from the filtrate by pouring the filtrate into an evaporating dish and evaporating it nearly to dryness. See Figure 1-14 for the correct setup.

**CAUTION** When using the burner, make certain that you confine loose clothing and that long hair is securely tied back. Wear your safety goggles, apron and gloves!

6. Remove the flame as soon as the liquid begins to spatter. Shut off the gas burner. What property of salt prevents it from being separated from the water by filtration?

7. At the end of this part of the experiment, all equipment you store in the lab locker or drawer should be completely cool, clean, dry, and arranged in an orderly fashion for the next lab experiment. Check to see that the valve on the gas jet is completely turned off. Make certain that the filter papers and sand are thrown into waste jars or containers and not down the sink! Wash your hands thoroughly before leaving the lab.

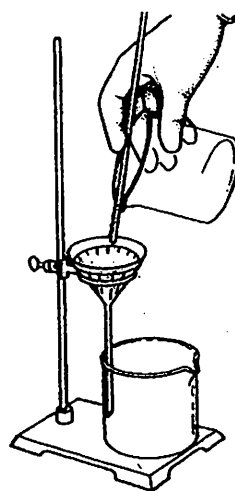
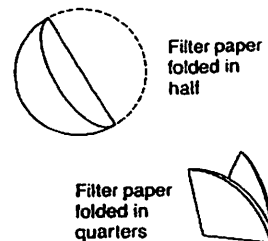


Figure 1-12



Filter paper ready for funnel

Filter paper in funnel

Figure 1-13

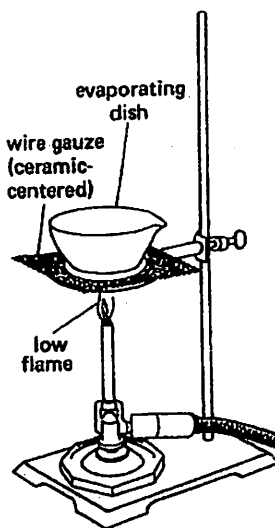


Figure 1-14

## Part 7 Measuring Temperature and Heat

### Apparatus

calorimeter (plastic cups and lid)  
ceramic-centered wire gauze  
burner and tubing  
Celsius thermometer  
beaker tongs  
iron ring

buret clamp  
graduated cylinder  
beaker, 250 mL  
sparker  
ring stand

### Materials

water

### Procedures

1. A thermometer is used to measure temperature and temperature changes. Examine your thermometer and the temperature range for the Celsius temperature scale. Compare the Celsius scale with the Fahrenheit scale as shown in Figure 1-15. What do you observe?

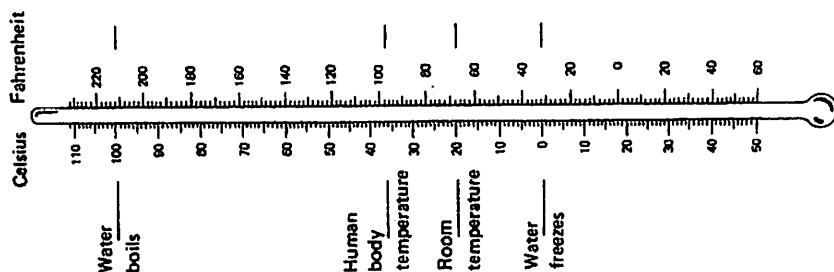


Figure 1-15

Name \_\_\_\_\_

**Lab 1- Evaporation and Filtration (Show All Work with Units)**

Exact mass of salt used

\_\_\_\_\_

Mass of empty evaporating dish

\_\_\_\_\_

Mass of evaporating dish with recovered salt

\_\_\_\_\_

Mass of recovered salt

\_\_\_\_\_

Calculate the percent salt recovered below.

Can you account for this percentage? Explain.

**Explain what would happen to the percentage of the salt recovered if:**

1. salt was left on the weigh paper after being transferred to the beaker?
2. the sand was still wet after decantation?
3. the filter paper had a hole in it?
4. carbon soot was deposited on the bottom of the evaporating dish from incomplete combustion of propane?

# Quantitative Observations of a Chemical Reaction

## Pre-Lab Discussion

Most experiments require the investigator to make some quantitative observations, or measurements. The numerical values of these measurements are called *data*. The most frequently measured quantities in the chemistry laboratory are mass, volume, and temperature.

When conducting an experiment of a quantitative nature, the first step in the procedure is to make and record measurements of the materials that are being investigated. If the materials take part in a chemical reaction (undergo chemical change), many, if not all, of the initial measured values probably will change. The nature and extent of these changes often help the investigator to understand what is taking place. Some of these changes, such as temperature change, can be measured and recorded as the reaction is taking place. When the reaction is ended, measurements again are made and recorded. The collected data from all of these measurements provide an overall record of what quantitative changes took place during the reaction.

When making measurements, you should keep in mind that the numerical values can be only as accurate as the instruments used to make the measurements. These values also are affected by the care and skill of the person using the instruments. As you gain more experience in the laboratory, you will become more familiar with the limitations and accuracy of the various instruments you use. You also will become more skillful in the use of these instruments and in carrying out various activities that are essential to a successful investigation.

Scientists must be imaginative. In many cases, they must devise their own experiments and decide what measurements will provide useful information. In this investigation, you will make measurements to determine the effects of a chemical reaction (combustion). You then will be asked to decide how these measurements can be used to extend your understanding of the reaction.

## Purpose

Make a quantitative investigation of a chemical reaction.

## Equipment

laboratory balance	graduated cylinder, 100-mL
ring stand	watch or clock with second hand
iron ring	glass square
wire gauze	safety goggles
thermometer	lab apron or coat
beaker, 250-mL	

## Materials

candle (2 cm diameter)      matches, 2 or 3

## Safety



In this experiment, you will be working with an open flame. Tie back long hair and secure loose clothing. Also, wear safety goggles and a lab apron or coat at all times when working in the lab. Be sure matches are completely extinguished before they are discarded.

## Procedure

1. Find the mass of the candle.
2. Measure exactly 100 mL of tap water in a graduated cylinder. Pour the water into a 250-mL beaker and place the beaker on wire gauze as shown in Figure 2-1. Measure the temperature of the water.

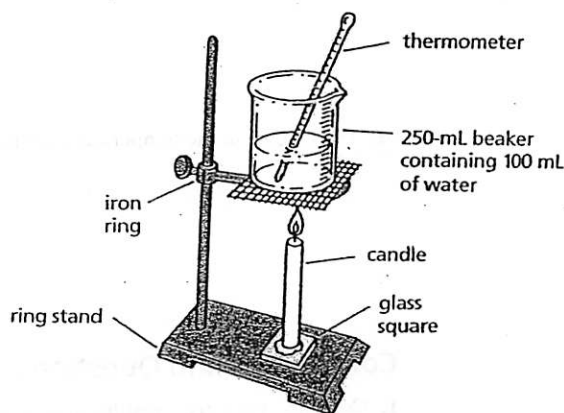


Figure 2-1



3. Light the candle and place it on the glass square, as shown. Adjust the height of the ring so that the flame is 2 cm below the base of the beaker. Using the candle, heat the water for exactly 10 minutes. Extinguish the flame and measure the temperature of the water and the mass of the candle.
4. Relight the candle and repeat steps 1–3 for a second trial.

## Observations and Data

	Trial 1	Trial 2
Original mass of candle	_____	_____
Mass of candle after burning	_____	_____
Time candle burned	_____	_____
Original temperature of water	_____	_____
Final temperature of water	_____	_____
Time water heated	_____	_____

## Calculations

For each trial, find:

	<u>Trial 1</u>	<u>Trial 2</u>
1. The change in the mass of the candle	_____	_____
2. The change in the mass of the candle per minute	_____	_____
3. The change in the temperature of the water	_____	_____
4. The change in the temperature of the water per minute	_____	_____

## Conclusions and Questions

1. Compare your trial results and calculations with those of other lab teams. Are your results exactly the same? How do you account for any differences in data? If one set of data differs from another in an experiment, does this mean that one or both sets are wrong? Explain your answer.

2. What does the term rate mean? a. What was the rate of burning of the candle? b. What was the rate of heating of the water?

3. Explain how the heat from the combustion reaction is related to the temperature change of the water.

4. Outline a laboratory procedure that would determine which produces more heat—a gram of candle wax or a gram of alcohol. How could this type of experiment be used to decide which substance would make the better fuel? What other factors might enter into choosing a fuel?